

WHAT IS CLAIMED IS:

1. A method of sintering a valve metal comprising sintering said valve metal in the presence of at least one iodine source to form a sintered valve metal.
2. The method of claim 1, wherein during said sintering, a valve metal-iodine compound forms along with said sintered valve metal.
3. The method of claim 1, wherein said iodine source is a gas.
4. The method of claim 1, wherein said iodine source is a liquid.
5. The method of claim 1, wherein said iodine source is a solid.
6. The method of claim 1, wherein said sintering occurs in a vacuum furnace or reactor.
7. The method of claim 1, wherein said sintering occurs in a vacuum furnace that has an isolatable trap.
8. The method of claim 2, further comprising collecting at least a portion of said valve metal-iodine compound in an isolatable trap for reuse.
9. The method of claim 1, wherein said valve metal is tantalum.
10. The method of claim 1, wherein said valve metal is niobium.
11. The method of claim 2, wherein said valve metal-iodine compound is tantalum iodide.
12. The method of claim 2, wherein said valve metal-iodine compound is TaI_5 or NbI_5 .
13. The method of claim 1, wherein said sintering is at a temperature of less than about 1200° C.
14. The method of claim 1, wherein said sintering is at a temperature of from about 350 to about 900° C.

15. The method of claim 1, wherein said sintering is at a temperature of from about 450 to about 850° C.

16. The method of claim 1, wherein said sintering is at a temperature in which the predominate sintering mechanisms comprise surface diffusion and evaporation/condensation.

17. The method of claim 1, wherein said sintering is for a time of from about 10 minutes to about 50 hours.

18. The method of claim 2, wherein said valve metal and said valve metal-iodine compound are present in equilibrium.

19. The method of claim 6, wherein said vacuum furnace further comprises an isolatable addition system for containing an oxygen getter.

20. The method of claim 6, further comprising deoxidizing said valve metal within said vacuum furnace.

21. The method of claim 1, wherein at least one oxygen getter is present during said sintering.

22. The method of claim 21, wherein said oxygen getter comprises magnesium.

23. The method of claim 1, further comprising deoxidizing before, during, and/or after said sintering.

24. The method of claim 23, wherein said deoxidizing is a magnesium deoxidizing.

25. A sintered valve metal formed by the method of claim 1.

26. A capacitor comprising the sintered valve metal of claim 25.

27. A method of forming a sintered valve metal, comprising:

sintering a valve metal in the presence of an iodine source within a container; and

deoxidizing said valve metal in the presence of an oxygen getter within said container.

28. The method of claim 27, wherein during said sintering, a valve-metal iodine compound forms along with said sintered valve metal.
29. The method of claim 27, wherein said iodine source is a gas.
30. The method of claim 27, wherein said iodine source is a liquid.
31. The method of claim 27, wherein said iodine source is a solid.
32. The method of claim 28, wherein said valve metal comprises tantalum, said valve metal-iodine compound comprises TaI_5 , and said oxygen getter is MgI_2 .
33. The method of claim 27, wherein said deoxidizing occurs before, during, and/or after said sintering.
34. The method of claim 27, wherein said container comprises a vacuum furnace.
35. The method of claim 34, wherein said container further comprises an isolatable trap.
36. The method of claim 35, further comprising collecting at least a portion of said valve metal-iodine compound and at least a portion of said oxygen getter in one or more isolatable traps for reuse.
37. The method of claim 34, wherein said container further comprises an isolatable addition system for containing said oxygen getter.
38. The method of claim 27, wherein said valve metal is tantalum.
39. The method of claim 27, wherein said valve metal is niobium.
40. The method of claim 27, wherein said valve metal has an oxide layer that is tantalum pentoxide or niobium pentoxide.
41. The method of claim 27, wherein said sintering is at a temperature of less than about 1200° C.

42. The method of claim 27, wherein said deoxidizing is at a temperature of less than about 1200° C.

43. The method of claim 27, wherein said sintering and said deoxidizing is at a temperature of from about 350 to about 900° C.

44. The method of claim 27, wherein said deoxidizing is at a temperature of from about 350 to about 900° C.

45. The method of claim 27, wherein said sintering is at a temperature of from about 450 to about 800° C.

46. The method of claim 27, wherein said deoxidizing is at a temperature of from about 450 to about 850° C.

47. The method of claim 27, wherein said sintering is at a temperature in which the predominate sintering mechanisms comprise surface diffusion and evaporation/condensation.

48. The method of claim 27, wherein said sintering is for a time of from about 2 to about 50 hours.

49. A sintered valve metal formed by the method of claim 27.

50. A capacitor comprising the sintered valve metal of claim 49.

51. The method of claim 28, wherein said valve metal comprises niobium, said valve-iodine compound comprises NbI_5 , and said oxygen getter is MgI_5 .

52. The method of claim 1, wherein said sintering occurs before any anodization.

53. The method of claim 1, wherein said sintering occurs after at least one anodization.

54. The method of claim 27, wherein said sintering or said deoxidizing or both occurs before any anodization.

55. The method of claim 27, wherein said sintering or said deoxidizing or both occurs after at least one anodization.

56. A method of making a capacitor comprising sintering a valve metal in the presence of an iodine source to form a sintered valve metal, and anodizing said sintered valve metal.

57. A valve metal powder, wherein when sintered at 800°C for 6 hours and formed in an anode with a formation voltage of 60 volts and a formation temperature of 83°C has a capacitance that is at least 20% greater than the same powder being tested and formed into an anode by sintering at 1400°C for 10 minutes at the same formation voltage and same formation temperature.

58. The valve metal powder of claim 57, wherein the DC leakage for said powder when sintered at 800°C and formed into an anode is 2.0 nA/CV or less.

59. The valve metal powder of claim 57, wherein said DC leakage for said powder sintered at 800°C and formed into an anode is 1.0 nA/CV or less.

60. The valve metal powder of claim 57, wherein said DC leakage for said powder sintered at 800°C and formed into an anode is 0.75 nA/CV or less.

61. The valve metal powder of claim 57, wherein said DC leakage for said powder sintered at 800°C and formed into an anode is 0.5 nA/CV or less.

62. The valve metal powder of claim 57, wherein said DC leakage for said powder sintered at 800°C and formed into an anode is 0.4 nA/CV or less.

63. The valve metal powder of claim 57, wherein said DC leakage for said powder sintered at 800°C and formed into an anode is 0.2 to about 1.0 nA/CV or less.

64. The valve metal powder of claim 57, wherein said valve metal powder is a tantalum powder.

65. The valve metal powder of claim 57, wherein said valve metal powder is niobium.

66. A valve metal powder wherein when sintered at 800°C for 6 hours and formed into an anode with a formation voltage of 60 volts and a formation temperature of 83°C has a DC

leakage that is at least 20% lower than the DC leakage obtained when the same powder is formed into an anode sintered at a temperature of 1400°C for 10 minutes at the same formation temperature and same formation voltage.

67. The valve metal powder of claim 66, wherein said DC leakage is at least 30% lower when sintered at 800°C.

68. The valve metal powder of claim 66, wherein said DC leakage is at least 40% lower when sintered at a temperature of 800°C.

69. The valve metal powder of claim 66, wherein said DC leakage is at least 50% lower when sintered at a temperature of 800°C or less.

70. The valve metal powder of claim 66, wherein said DC leakage is from about 20% to about 70% lower when sintered at a temperature of 800°C.

71. The valve metal powder of claim 57, wherein said capacitance is at least 30% greater when sintered at 800°C.

72. The valve metal powder of claim 57, wherein said capacitance is at least 40% greater when sintered at 800°C.

73. The valve metal powder of claim 57, wherein said capacitance is at least 20% to about 70% greater when sintered at 800°C.

74. The valve metal powder of claim 57, wherein said capacitance is from about 40% to about 60% greater when sintered at 800°C.

75. A sintered valve metal body having a shrinkage diameter of 0.5% or less with an initial press density of 5.5 g/cc.

76. The sintered valve metal body of claim 75, wherein said shrinkage is 0.25% or less.

77. The sintered valve metal body of claim 75, wherein said shrinkage is about 0% or

less.

78. The sintered valve metal body of claim 75, wherein said shrinkage is -0.5% or less.
79. The sintered valve metal body of claim 75, wherein said shrinkage is -0.75% to -5.0%.
80. The sintered valve metal body of claim 75, wherein said shrinkage is from 0.5% to -1.0%.
81. The sintered valve metal body of claim 75, wherein said sintered body has a DC leakage of 2.0 nA/CV or less when formed into an anode with a formation voltage of 60 volts and a formation temperature of 83°C.
82. The sintered valve metal body of claim 81, wherein said DC leakage is 1.0 nA/CV or less.
83. The sintered valve metal body of claim 81, wherein said DC leakage is 0.75 nA/CV or less.
84. The sintered valve metal body of claim 81, wherein said sintered body has a capacitance of at least 30,000 CV/g under the same conditions.
85. A sintered valve metal body, that when formed into an anode by sintering at 800°C for 6 hours has a DC leakage of 2.0 nA/CV or less, using a formation voltage of 60 volts and a formation temperature of 83°C.
86. The sintered valve metal body of claim 86, wherein said DC leakage is 1.0 nA/CV or less.
87. The sintered valve metal body of claim 86, wherein said DC leakage is 0.5 nA/CV or less.
88. A sintered valve metal body which, when formed into an anode sintering at 800°C for 6 hours with a formation voltage of 60 volts and a formation temperature of 83°C has a

capacitance of at least 40,000 CV/g.

89. The sintered valve metal body of claim 89, wherein said capacitance is from 40,000 to about 250,000 CV/g.

90. A method of making a capacitor anode comprising pressing a basic lot valve metal powder into a green anode and sintering said green anode to form a capacitor anode, without a separate deoxidation step and without heat treating said basic lot valve metal powder prior to pressing into said green anode, and without any other thermal processing step.